Experimental investigation of the natural and forced convection on solidification of Sn-3wt. %Pb alloy using a benchmark experiment

Lakhdar Hachani, Redouane Boussaa, Bachir Saadi, X.D. Wang, Kader Zaidat, Aissaa Belgacem Bouzida, Dominique Henry, Val éry Botton, H. Ben Hadid, Yves Fautrelle

Experimental investigation of the natural and forced convection on solidification of Sn-3wt. %Pb alloy using a benchmark experiment

L. HACHANI[1,2,4], R. BOUSSAA[1,3], B. SAADI[1], X-D. WANG[1], K. ZAIDAT[1], A. BELGACEM BOUZIDA[2], D. HENRY[3], V. BOTTON[3], H. BEN HADID[3] and Y. FAUTRELLE[1]

1. Grenoble Institute of Technology, SIMAP-EPM Laboratory, PHELMA BP75 38402 Saint Martin d'Hères Cedex, France
2. Laboratory of Mechanics, University Amar Telidji of Laghouat, Algeria
3. LMFA, Ecole Centrale de Lyon, 36 avenue Guy de Collongue, 69134 Ecully Cedex, France
4. University Hadj Lakhdar of Batna, Algeria

ABSTRACT: We deal with the development of a solidification benchmark experiment in order to investigate the structure formation as well as solute macro- mesosegregation, by means of a well-controlled solidification experiment. The experiment consists in solidifying a rectangular ingot of Sn-3wt.%Pb alloy, by using two lateral heat exchangers which allow extraction of the heat flux from one or two vertical sides of the sample. The domain is a quasi two dimensional parallepipedic ingot (100x60x10) mm. The temperature difference ΔT between the two lateral sides is 40 K and the cooling rate CR = 0.03 K/s. The instrumentation consists in recording the instantaneous temperature maps by means of an array of 50 thermocouples in order to provide the time evolution of the isotherms. After each experiment the patterns of the segregations have been obtained by X-ray radiograph and confirmed by eutectic fraction measurements. The local solute distribution determined by means of induction coupled plasma analysis is provided. The originality of the present study is to examine the effect of the forced convection driven by a travelling magnetic field (TMF) induced by a linear inductor located on the bottom part of the sample. A periodically reversed stirring with a modulation frequency equal to 0.5 Hz stirring have been investigated. This study allows us to evaluate the evolution due to the forced convection induced by a TMF field, as well as its influence on the initial conditions, the solidification macrostructure and the segregation behavior. Measurements of the velocity field by ultrasonic Doppler velocimetry (UDV) method in a Ga-In-Sn pool were performed and transposed to the tin-lead alloy case before solidification. Post-mortem patterns of the macro-mesosegregations have been obtained by X-ray radiography. The results show the transport effects of the flow on both the macrosegregations and the channel formation. The reversal of the TMF produces a decrease of the level of mesosegregations, namely channel formation.

1. Experimental setup

The properties of most alloys depend largely upon the degree of control, which can be applied during the solidification process. One of the major problems lies in the non-uniform distribution of solute concentration that is inherent to solidification. These concentration variations, also known as segregations, appear at the mesoscopic scale (freckles) and/or at the ingot scale (macrosegregation) [2-3] in a solidified sample. Naturally these concentration variations have important repercussions on materials properties; therefore, minimizing segregation is essential for the proper alloys performance. It appears that the role of the gravity may be prominent for the evolution of segregation and can create unexpected concentration patterns. One of the possibilities to counter this effect is to impose a forced convection.

The introduction of magnetic field in the solidification process is one of the effective methods to improve the microstructure and mechanical performance of alloys [4-7]

In this study, the model alloy (Sn-3-wt.%Pb) was chosen as the experimental study subject on account of its low melting point. Based on the experiment result, we examine the effect of the forced convection driven by a travelling magnetic field on the solidification process in terms of temperature history, velocity field, structure maps and solute distribution in the solid sample during the horizontal directional solidification.

The experiment principle is similar, to the well-known Hebditch and Hunt experiment [3], with a special emphasis on controlling initial and thermal boundary conditions, and obtaining reproducible quantitative measurements [1].

In this study a periodically reversed stirring with a modulation frequency equal to 0.5 Hz has been investigated. The present work is aimed at evaluating:

1. How the fluid flow in the mushy zone transports the solute and generates segregations.
2. Whether it is possible to control the fluid flow by mean of an electromagnetic stirring device and accordingly to control segregation.

In the next section, the experimental process is detailled. The heat transfer and the macro-segregation are discussed. The description of the experimental device represented on Fig. 1, and process are presented elsewhere with more details in the work [7]. However in this experimental study we are interested primarily on the part of forced convection driven by travelling magnetic field stirring and its influence on the solidification process.

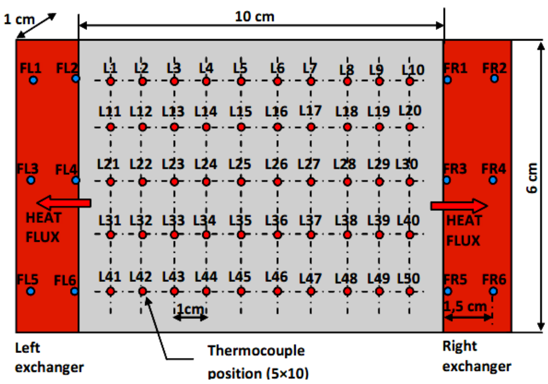


Fig.1. Sketch of the sample surrounded by the two lateral heat exchangers. The location of the lateral thermocouple lattice (L1 to L50) welded on one of the largest surface of the crucible is also shown.

The travelling magnetic field is generated by linear motor placed underneath the bottom wall of stainless steel crucible. The corresponding electromagnetic force is imposed from the melting to solidifying steps. Two experiments have been investigated:

Case I, the melt is solidifying under the natural convection. The temperature difference between the two lateral sides of the sample produces clockwise fluid flow recirculations.

Case II the experimental conditions are the same as case I, but the electromagnetic force direction is periodically reversed. The modulation frequency is equal to 0.5 Hz.

2. Experimental result

2.1 *Magnetic fields evolution*

The linear inductor has eight poles and is located underneath the bottom wall of the crucible about 5 mm. Its length *is* L_{ind} = *244 mm*. This inductor is fed by a three-phase AC current of normal frequency ($f_0 = 50Hz$). The wave-length l and the pole pitch τ are $l = 2\tau = 48$ *mm*, so that the wave number is $k = 2\pi/l = 130.899\ m^{-1}$. The synchronism velocity of the moving magnetic field is $U_S = f_e l = \omega_0 / k = 2.4 m/s$.

A measure of the modulus of the magnetic field $\|\vec{B}\| = \sqrt{B_x^2 + B_y^2}$ in different *x*-positions in the absence of liquid metal for various applied current (*7.5*, *8.5* and *10 A*) at a frequency of 50 *Hz* as show that for an intensity rating of 8.5A the magnetic field strength is about 50 *mT*.

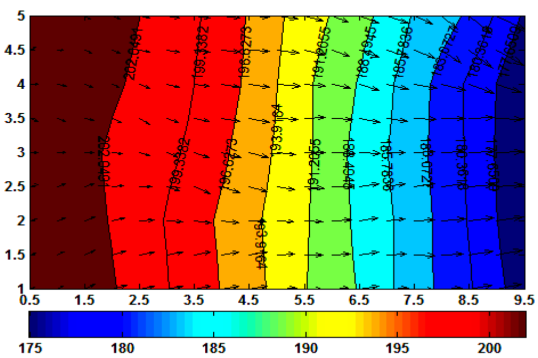
2.2 *Velocity measurement by ultrasonic Doppler velocimetry (UDV)*

In order to assess the flow characteristics before solidification, velocity measurements were performed with the eutectic (GaInSn: 67*wt*.%Ga-20.5*wt*.%Sn-12.5*wt*.%In) alloy. The liquid metal, whose density is 6400 kg/m^3, and dynamic viscosity 2.16×10^{-3} Pa.s [8], is contained in a transparent Plexiglas rectangular tank of width *W*=1cm, length *L*=10cm, and height *H*=6cm. An Ultrasonic Doppler Velocimetry (UDV) sensor is fixed on a vertical sidewall of the tank to probe the horizontal velocity components. The data provided by the ultrasonic velocimetry UDV consist in instantaneous velocities in the *x* direction V_x (x, t_i). The results show that for applied current I = 7.5*A* the mean (time averaged) velocity $\overline{V_x}$ varies over a large range from 17.96 to 34 *mm/s* and exhibits significant fluctuations around 25.19 *mm/s*, and the corresponding Reynolds number is ($\overline{\mathrm{Re}} = 44455$). It is very important to note that all experimental measurements performed on (GaInSn), are achieved in order to estimate the configuration dynamics in the melt of Sn-03*wt*.%Pb with an approximation by taking into account the similarity coefficient

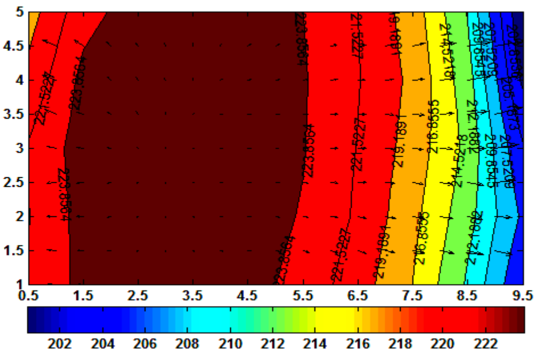
$$\frac{velocity(Sn-Pb)}{velocity(Ga-In-Sn)} = \sqrt{\frac{\dfrac{\rho(Sn-Pb)}{\sigma(Sn-Pb)}}{\dfrac{\rho(Ga-In-Sn)}{\sigma(Ga-In-Sn)}}} = 0.4294$$

2.1 *Temperature fields and thermal gradient evolution*

In the following subsection, we show in Fig. 2 temperature contours coupled with thermal gradient for two cases of solidification discussed previously at selected moment (*t* = 14740 s) during the cooling. The experimental conditions are: *ΔT* = 40 K, CR = 0.03 K/s. At the initial stage the temperature difference between the two lateral sides of the heat exchangers the left wall temperature is T_L = 280 °C and the right one T_R = 240 °C. The magnetic force with the applied current $I = 9.56\ A$, is imposed on liquid Sn-Pb alloy (electrical conductivity σ = 3.86x10^6 Ω^{-1}.m^{-1}). The temperature gradients were calculated at each thermocouple location from the temperature difference between two adjacent points by means of a numerical centered difference scheme.



Case-I: Tmax = 203.43°C; Tmin = 173.67 °C
ΔT = 29.76 °C



Case-II: Tmax = 226.19 °C; Tmin = 200.52 °C
ΔT = 25.67 °C

Fig.2. Temperature field maps selected at time (t = 14740s), for the two cases of solidification (I and IV). The experimental conditions: Sn-3wt.%Pb, ΔT = 40 K, CR = 0.03 K/s, applied current *I* = *9.56 A*, f = 50 Hz, σ = 1.86x10^{6} Ω^{-1}.m^{-1}, B$_0$ = 30 mT, liquidus temperature T_l = 227.7°C, eutectic temperature T_s = 183°C. The arrows correspond to the $-\nabla T$ vector which is proportional to the local heat flux density

2.3 *Velocity estimate*

The temperature data are analyzed to obtain some clues as to the magnitude of the velocity field during solidification. The method consists in calculating the projection of the velocity field on the temperature gradient vector at each node corresponding to the location of a thermocouple. The principle of the method is presented with more details in L. Hachani et al [7].The algebraic velocity estimate U is defined as $U=\vec{U}.\nabla T/(|\nabla T|)$ where $|\nabla T|$ is the norm of the temperature gradient. Figure (3) shows the time evolution of the velocity projections on the thermal gradient vector for various internal nodes L14, L15, L16, L17 and L18 (shown in Fig.1) located in the upper part of the ingot where the scalar products of the velocity vector $\vec{u}$ and the temperature gradient ∇T are supposed not to be too small.

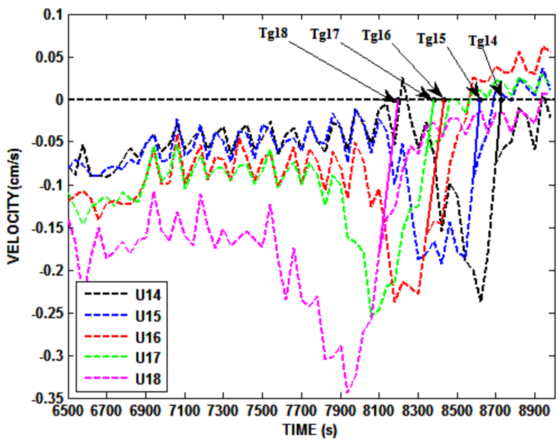


Fig.3. Velocity estimate U versus time for various internal nodes (L14, L15, L16, L17 and L18) located in the upper part of the ingot. Fig. 5 also shows the extrapolation to zero used to estimate the instant when the columnar front crosses the node position

Various observations may be drawn from such analysis. Firstly, we retrieve the various steps of the solidification, namely the global stabilization of the velocity during the thermal natural convection regime before cooling. Secondly the velocity decays when the columnar front becomes close to the node.

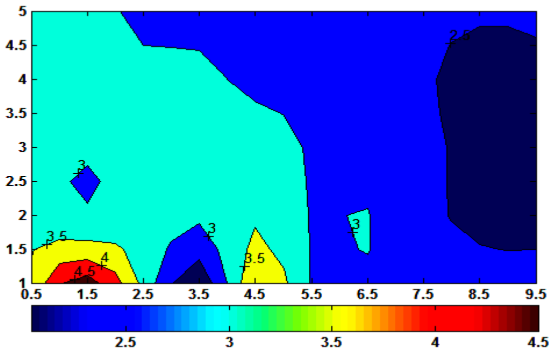
3. Post-mortem analysis

3.1 *Concentration map for Sn-3wt.%Pb alloy*

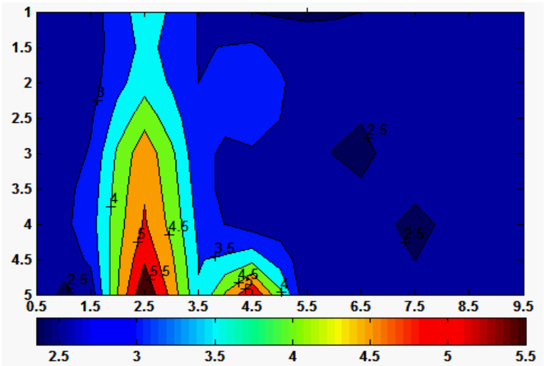
A chemical method coupled to ICP is used to analyze quantitatively the lead concentration distribution. We drilled fifty holes into ingot to extract about 200 mg for each analyzed sample. The holes positions which correspond to the thermocouple positions are presented in Fig. 1. The results illustrated by Fig. 4, show the segregation distribution for the two cases of solidification for identical experimental conditions (Sn-3wt.%Pb alloy. *DT* = *400K/m*, *CR* = *0.03K*/s). We have checked that the measured average concentration was close to the nominal one.

3.2 *X-Ray analysis*

X-Ray radiographs of the samples were performed by CEA-Saclay laboratory (France) (Fig. 5) in order to analyze qualitatively the lead concentration distribution. The main objective is to observe all types of macro-meso segregations, specially the channel distributions. The lead easily absorbs the X-rays, and the color contrast gives indications of the solute distribution within the ingot. Lead-rich solute zone is easily visible and appears in the photography.



Case I: Min = 2.05%; Max = 4.74%; Aver = 2.95%



Case II: Min = 2.35 %; Max = 6 %; Aver = 3.03%

Fig.4. Lead concentration for Sn-3wt.%Pb. Applied temperature difference ΔT = 40 K. Cooling rates 0.03 K/s. Two cases are presented: case I natural convection and case II natural and modulated forces (modulation frequency *f = 0.5 Hz*), *I = 9.56 A*.

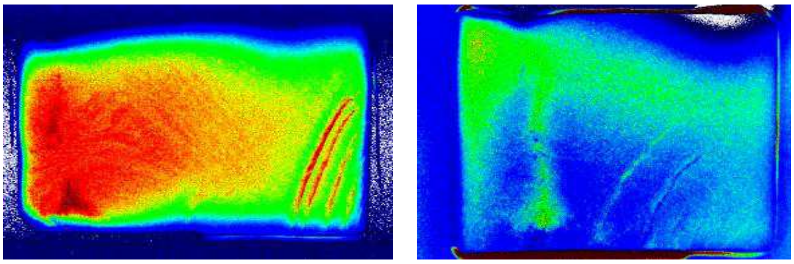


Fig.5. X-ray radiograph of the solidified ingot showing the freckles, Sn-3wt.%Pb alloy. DT = 400K/m, CR = 0.03K/s. left natural convection, right modulated forces (modulation frequency f=0.5 Hz), ***I = 9.56 A*****.**

The modulated electromagnetic stirring has shown its effectiveness, particularly in the significant reduction of the number of segregated channel but doesn't suppress them completely. The forced convection changes also the channel locations

Conclusion

This work constitutes an experimental study of the effect of natural and forced convections in a benchmark solidification experiment using Sn-wt.3%Pb alloys. The thermal phenomena, grain structure and macro- mesosegregation (freckles) were examined. The role of melt convection has been studied during solidification. The melt was electromagnetically agitated by means of a travelling magnetic field (TMF) of moderate intensity. A periodically reversed stirring with a modulation frequency equal to 0.5 Hz stirring has been investigated. The following major conclusions can be drawn: The forced convection influences significantly the macrostructure and segregation distribution.The effect of the electromagnetic stirring on the flow pattern in the molten metal is very important. The thermal process quantities, namely temperature gradients, cooling rate at the solidifying front and liquidus interface velocity, can be used to characterize the morphology of the grain structures.

Acknowledgments

The authors acknowledge the European Space Agency through the CETSOL project (ESA-MAP AO-99-117) as well as the SMACS ANR project for their financial support. The authors are indebted to B. Rattoni (CEA-Saclay, France) who performed the X-ray characterizations.

References

[1] X.D. Wang and Y. Fautrelle, An investigation of the influence of natural convection on tin solidification using a quasi two-dimensional experimental benchmark, Int. J. Heat and Mass Transfer V. 52, (2009), 5624-5633.

[2] Sarazin, J. R., Hellawell, A Channel Formation in Pb-Sn, Pb-Sb, and Pb-Sn,Sb Alloy Ingots and comparison with the system *NH4 Cl-H2O*. Metall Met Trans A, V. 19, (1988), 1861-1871

[3] D.J. Hebditch, J.D. Hunt, Observations of ingot macrosegregation on model systems, Metallurgical transactions, V. 5, (1974), 1557-1563.

[4] Campanella T, Charbon C, Rappaz M. Grain refinement induced by electromagnetic stirring: A dendrite fragmentation criterion. Metall Mater Trans A, V. 35, (2004), 3201-3210.

[5] Zaïdat K, Mangelinck-Noël N, Moreau R. Control of melt convection by a traveling magnetic field during the directional solidification of Al-Ni alloys. CR Mecanique, V. 335 (2007), 330-335.

[6] Willers B, Eckert S, Michel U, et al. The columnar to equiaxed transition in Pub-Sn alloys affected by electromagnetically driven convection. Mater Sci Eng A, V. 402, (2005), 55-65.

[7] Lakhdar. HACHANI & al. Experimental analysis of the solidification of Sn-3 wt.%Pb alloy under natural convection. Int. J. of Heat and Mass Transfer V. 55, (2012), 1986-1996.

[8] Xiaodong Wang, & al. A periodically reversed flow driven by a modulatedtraveling magnetic field. Part I: Experiments with GaInSn. Metallurgical and Materials Transactions B. V. 40, (2009), 82-90.